

ERRATA

SP-191 Water-Cement Ratio and Other Durability Parameters

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Determination of Water-Cement Ratio in Hardened Concrete by Optical Fluorescence Microscopy, by U. H. Jakobsen, P. Laugesen, and N. Thaulow

The synopsis as printed is incorrect. The correct synopsis is as follows:

Synopsis: This paper describes a method for determining the water to cement ratio (w/c) of hardened concrete using optical fluorescence microscopy. The method is well established and has been used for many years. In Denmark the method is used for quality control of hardened concrete. The method is based on vacuum impregnation of concrete using a yellow fluorescent epoxy. During impregnation the capillary porosity, cracks, voids, and defects in the concrete are filled with epoxy. The amount of fluorescent dye entering the cement paste depends on the capillary porosity, which is determined by the w/c and the degree of hydration. After impregnation and hardening of the epoxy a thin section of concrete with a thickness of 0.020 mm (20 μm) is prepared. The thin section is analyzed under an optical microscope using a combination of a blue excitation filter and a yellow blocking filter. This is the fluorescent light mode in which epoxy filling air voids and cracks appears yellow, cement paste as shades of green, and aggregate black. The shade of green of the cement paste depends on the capillary porosity. A sample with low w/c appears dark green, i.e. has less fluorescence intensity due to a low amount of epoxy within the paste. A sample with high w/c appears light green, i.e. has high fluorescence intensity. These shades of green (fluorescence intensity) are used to determine the w/c by comparing the fluorescence intensity of the cement paste with standards of known w/c . This paper describes the fluorescent impregnation technique, the thin section preparation, the visual determination of w/c and discusses the pitfalls in the w/c determination. Furthermore, the paper presents data from a quality assurance project and damage analysis and data of Round Robin Testing.

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Water-Cement Ratio and Other Durability Parameters

Techniques for Determination



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Water-Cement Ratio and Other Durability Parameters— Techniques for Determination

Editor

Mohammad S. Khan



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PREFACE

This volume contains eight of the 12 papers that were presented in a two-part session "Methods for Determining Water-Cement Ratio and Other Durability Parameters in Concrete," sponsored by ACI Committee 201, Durability of Concrete, at the 1998 Fall Convention in Los Angeles. I express my appreciation and thanks to Steve Forster and Gary Crawford, both of the Federal Highway Administration, who co-chaired the sessions with me.

The authors who made presentations and contributed to this publication should be commended for coming forward and sharing their knowledge and experience with their peers on many techniques that are not standardized yet and are still in the developmental stage. The efforts of the reviewers, many of them members of Committee 201, are equally commended for their constructive comments and suggestions that would contribute to refinement and maturity of several techniques included in this publication. On behalf of Committee 201, I thank all the authors and reviewers.

This publication should be of interest to individuals involved in concrete failure investigations, particularly those related to durability, and in quality control efforts aimed at assuring a durable structure. Academics, researchers, materials engineers, structural engineers, forensic engineers, and materials producers should all benefit from the information presented in this publication.

Editor

Mohammad S. Khan

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Concrete Durability—Influencing Factors and Testing

by S. W. Forster

SYNOPSIS

Durability is defined in ACI 116R as the ability of concrete to resist weathering action, chemical attack, abrasion, and other conditions of service. Concrete can certainly be used to construct durable, long-lasting pavements and structures; we see numerous examples of this behavior daily as we go about our lives. Durability remains an issue, however, because we also see some examples of concrete construction that have not been as distress-free, or lasted as long as we would have liked. In these latter instances, usually one or more aspects of the environment, materials and mix design and/or construction were not sufficiently considered for the impact they would have on the performance of the concrete used. The objective of this paper is to review the various aspects of concrete durability as considered by ACI Committee 201, particularly as to the demands placed on the concrete as a material. Current methods used to explain the durability performance of in-service concrete or predict the performance of concrete to be placed will also be reviewed.

The aspects of durability that affect and interact to produce the durability performance of concrete may be placed in five broad categories: freezing and thawing; aggressive chemical attack; surface abrasion; corrosion of embedded steel; and the alkali-aggregate reaction. Tests used to estimate concrete durability include microscopic examination, chemical techniques, characterization of the concrete components, and accelerated testing to simulate the durability aspect under consideration.

Interpretation of the performance of in-service concrete is made more difficult by the fact that usually the deterioration is not caused by one type of distress, but involves a combination of factors.

Keywords: alkali-aggregate reaction; chemical attack; concrete durability; corrosion; freezing and thawing resistance

Stephen W. Forster is a Research Geologist and Team Leader of the Portland Cement Concrete Pavement Team in the Office of Infrastructure Research and Development of the Federal Highway Administration (FHWA), Washington, D.C. Dr. Forster is currently in charge of the research program on all aspects of concrete pavements, including the durability of the materials used.

INTRODUCTION

Durability is defined in ACI 116R (1) as the ability of concrete to resist weathering action, chemical attack, abrasion, and other conditions of service. Durability requirements according to the ACI building code are found in ACI 318/318R, Chapter four (2). The mission of ACI Committee 201, Durability of Concrete, is “to develop and report information to ensure the production of durable concrete”(3). The factors that influence durability, as addressed by ACI 201, will be considered herein.

Concrete can certainly be a durable, long-lasting construction material; we see numerous examples of durable concrete in our daily lives. Durability remains an issue, however, because we also see some examples of concrete construction that have not been as distress-free, or lasted as long as they were designed to do. In these latter instances, usually one or more aspects of the environment, materials and mix design and/or the construction process were not sufficiently considered for the impact they would have on the performance of the concrete used. The objective of this paper is to review the various factors of concrete durability that may influence the long term performance of concrete in service, particularly as to the demands placed on the concrete as a material, and review the methods currently used to test and evaluate the durability performance of in-service concrete or predict the performance of concrete prior to placement.

For the purposes of this paper, durability and deterioration will be limited to the following five main factors: freezing and thawing; aggressive chemical attack; surface abrasion; corrosion of embedded steel; and alkali-aggregate reaction. Concrete can range from very resistant to very susceptible to each of these deterioration mechanisms, depending on the characteristics of the concrete and the exposure conditions. In the following sections each of these factors of durability and the means of testing and evaluating them will be discussed.

FACTORS INFLUENCING DURABILITY

Freezing and Thawing

The resistance of concrete to deterioration by freeze-thaw cycling can potentially involve two different components of the concrete: the cement paste and the aggregate. In order for freezing and thawing to have an effect on either component, however, they must be at some level of critical saturation at the time of freezing.

In spite of extensive study and research on the subject, disagreement still exists

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as to the exact mechanism of stress development in the cement paste due to freezing while at a critical level of saturation. Early theories ascribed the damage to hydraulic pressures in the pores, generated by the resistance to water movement away from the area of freezing within the paste. The magnitude of the pressure was thought to be related to the rate of freezing, with higher freezing rates generating greater pressures. The pressure was also believed to depend on the permeability of the paste and the distance necessary for the water to travel to reach a point of "escape" or pressure relief. Thus, the beneficial effect of entraining air in the concrete was explained as decreasing the distance necessary for the water to travel in order for an air void (site of pressure relief) to be encountered. Later theories related the phenomenon to osmotic potential created as a result of increases in alkali concentrations in the unfrozen portion of the pore solution during freezing. This potential causes the water in the unfrozen pores to diffuse into the solution in the frozen pores. The dilution of water in the vicinity of ice allows additional ice formation (or ice accretion). This in turn can cause dilation pressure in the paste. In this scenario the entrained air voids serve as a preferred site for the unfrozen water, thus avoiding the distress due to the osmotic mechanism.

The effect of freezing and thawing on certain aggregates in concrete has to do with the porosity, pore size distribution and permeability of the susceptible aggregates. The hydraulic pressure theory, described above for cement paste, may be the best explanation for the behavior of these aggregates. In general, it is the coarse aggregate fraction that causes the distress in concrete. The susceptible aggregates typically have high porosity and absorption, with a majority of pores in the 0.1 to 5 Fm range. Pores in this size range can become saturated so that excessive pressures and cracking result during freezing. Large pores do not usually become completely filled with water, whereas any water in pores finer than the designated range usually does not freeze. Susceptible aggregates can lead to popouts near the surface of concrete. In concrete located in climates subject to freezing and thawing, these aggregates cause the phenomenon known as D-cracking. In this situation, the concrete near free edges and joints is exposed to sufficient water for the aggregate to become critically saturated. Upon freezing, expansion of the aggregate, and/or rapid expulsion of the water in the aggregate into the paste, cause disruptive pressures and cracking in the concrete parallel to joints and free edges. Experience has shown that reduction in maximum aggregate size improves performance for the susceptible aggregates.

Freeze-Thaw Durability

There are a number of recommendations for achieving concrete resistant to deterioration due to freezing and thawing. These include minimizing the exposure to moisture; a low water-cementitious materials ratio; an adequate air void system; durable aggregates; adequate curing prior to exposure to the first freezing cycle; and good construction practices.

Since the susceptibility of concrete to freeze-thaw deterioration depends on the degree of saturation, minimizing the exposure to moisture and moisture uptake will be very beneficial, where practical. Where lack of critical saturation is a

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certainty, additional specific steps to prevent damage due to freezing and thawing will not be necessary. In those structures where saturation is a potential problem, further steps must be taken. It is recommended that the water-cementitious materials ratio be limited to certain values depending on the structure and exposure conditions to be encountered. See the 201.2R Guide to Durable Concrete (3) for more details. The concrete should contain an adequate system of entrained air voids so that the paste is protected during freeze-thaw cycling. Although these values are often given in terms of percentage of air in the concrete, it is preferable to express it as the air void spacing factor, which is more representative of the protection afforded by the air void system. This number should be 0.20 mm (0.008 in) or less. There is some indication that higher strength, lower permeability concretes can perform well with air void systems with greater spacing factors, but specific recommendations are still in the development stage. Aggregates should be of known durability, or if potentially susceptible, they should be tested for their freeze-thaw durability in concrete at the maximum particle size proposed for use. A special aspect of damage due to freezing and thawing is the early-age exposure of the concrete to freezing. Minimum strength levels are recommended to assure the concrete will be resistant to the stresses due to freezing at an early age. The required strength will depend on the degree of saturation of the concrete at the time of first freezing. Concrete exposed to deicing chemicals, particularly that in pavements, bridges and parking structures, should receive special care during construction. This care should include adequate consolidation; no overworking or over finishing of the surface; and no addition of water to aid in finishing.

Testing and Evaluation

There are a number of methods available to evaluate the resistance of concrete to distress due to freeze-thaw cycling. The tests and interpretation should be based on the recommendations given above. Water-cementitious ratios can be evaluated from the batching proportions for new concrete or by chemical and microscopic techniques for hardened concrete. Similarly, strength can be measured directly or estimated by various indirect non-destructive techniques. The amount of entrained air in the concrete may be estimated at the time of placement by measuring the total air content in the plastic concrete. A better indication of the adequacy of the entrained air system is gained by measuring the characteristics of the air void system in hardened concrete using ASTM C457, Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete (4). There are several tests used to evaluate the pore system in coarse aggregate as an indicator of its susceptibility to freeze-thaw cycling. The newest test is the hydraulic fracture test developed under the Strategic Highway Research Program. This test is still undergoing refinement. The concrete including the aggregate can be tested for freeze-thaw durability using ASTM C666, Resistance of Concrete to Rapid Freezing and Thawing (5). Adequacy of curing and consolidation, and results of finishing can be evaluated by using ASTM C856, Petrographic Examination of Hardened Concrete (6).

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Aggressive Chemical Attack

There are certain chemicals which in solution will readily attack concrete. Although the list is extensive, those usually encountered by common structures are fairly few. These include sulfates, sea-water, and carbon dioxide. Sea-water exposure causes a form of sulfate attack and increased corrosion potential for any reinforcing steel.

Sulfate Attack

Sulfate can occur in soils as compounds of sodium, calcium, magnesium and potassium. This is a potential problem when a concrete structure is placed in or on soils containing these compounds. These sulfates have the potential for accumulation at an exposed face of the concrete when evaporation (and therefore movement of groundwater through the concrete) from that face is taking place.

There are several chemical mechanisms involved in sulfate attack, both of which result in a volume increase. One involves the formation of gypsum from the sulfates and calcium from the cement hydration; the other entails the formation of ettringite from the sulfate and hydrated calcium aluminate. The latter reaction is thought to be the cause of most disruption caused by sulfate attack. The reaction of concrete with the sulfate ions in seawater is similar to that from sulfate attack related to soils, but is not as severe due to the influence of other ions present in sea-water. Recommendations to prevent sulfate attack include the use of a dense, low permeability concrete, and therefore a low water-cementitious materials ratio is beneficial. Also, there are several cement types specifically formulated to resist sulfate attack. These are Type II (moderate sulfate resistant) and Type V (sulfate resistant) cements. Both types have a limit on tricalcium aluminate content. Certain pozzolans and ground granulated blast furnace slag added to the concrete mix will also increase resistance to sulfate attack.

In addition to the tests for water-cementitious ratio, strength, and petrographic examination mentioned above, there has been some use of ASTM C1012, Length Change of Hydraulic-Cement Mortars Exposed to a Mixed Sodium and Magnesium Sulfate Solution (7), as an evaluation tool.

Carbonation

Carbonation of concrete is caused by exposure to carbon dioxide in the atmosphere or contained in groundwater. The results of the reaction are the production of carbonates and a net volume decrease of the concrete. The carbonated concrete is more susceptible to shrinkage and cracking, and therefore subject to increased moisture transport. Carbonation is usually a slow process, which works its way into the concrete from exposed surfaces. The rate of the reaction is tied to the relative humidity of the air in contact with the concrete, with highest rates occurring in the 50 to 75 percent range (3). As carbonation advances, it reduces the protection from corrosion the concrete cover and high pH affords any included steel.

It is recommended that the concrete be dense, well consolidated, and have low permeability to slow carbonation to an acceptable level. Tests for these

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characteristics, as well as water-cementitious materials ratio, can be run. In addition to petrographic examination, the existing concrete can be tested for depth of carbonation using phenolphthalein solution.

Surface Abrasion

Abrasion refers to the wearing away of the surface of concrete. It includes wear due to traffic, such as vehicles and pedestrians, and wear due to flow of material over the concrete, such as wind or water.

Abrasion due to traffic can be relatively minor for vehicles with plain (un-studded) tires and little or no turning and stopping actions. On the other hand, it can be high if studded tires or chains are used and there are both turning and braking actions as well as tire slippage. Abrasion due to water or wind depends a great deal on the particle load the medium is carrying and the velocity.

Abrasion resistance has been found to correlate well with concrete strength and density (thus water-cementitious materials ratio) as well as the type (hardness), maximum size and volume percentage of coarse aggregate exposed at the surface. In addition to testing for these characteristics, there are several concrete abrasion tests in ASTM. These include ASTM C1138, Abrasion Resistance of Concrete by Underwater Method (8); C418, Abrasion Resistance of Concrete by Air-Driven Silica Sand Impingement (9); C944, Abrasion Resistance of Concrete/Mortar Surfaces by Rotating Cutter (10); and C779, Abrasion Resistance of Horizontal Concrete Surfaces (11).

Corrosion

Under normal conditions, the alkaline environment provided by the pH in concrete provides protection against corrosion of the included reinforcing or prestressing steel. The longevity of that protection is dependant on the amount of concrete cover over the steel, the permeability of the concrete, and the exposure to which the concrete is subjected. A great deal of literature has been written on corrosion of steel in concrete, the reader is referred to these documents, for instance ACI 222R, Corrosion of Metals in Concrete (12), for detailed information.

Carbonation or other factors can lower the concrete pH to levels where corrosion can occur in the presence of moisture and oxygen. In addition, the penetration of chlorides from deicer application or other sources to the level of the reinforcing can initiate or accelerate corrosion. Conversely, in order to prevent corrosion, the pH should be maintained at a high level, chloride exposure kept low, and rate of chloride penetration into the concrete also kept low. As with a number of the other durability factors, this involves using a concrete that is dense, with low permeability (low water-cementitious materials ratio) and has sufficient cover over any included steel. Tests for these characteristics can be run, but the test most commonly used to evaluate the corrosion protection provided by a concrete is ASTM C1202, Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration(13), or some variation thereof. Interpretation of the test should be on a semi-quantitative basis rather than strict numerical comparisons.

Alkali-Aggregate Reaction

Alkali-aggregate reaction (AAR) is divided into two categories, alkali-silica reaction (ASR) and alkali-carbonate reaction (ACR).

ASR was first recognized in California in the 1940's, where it was noted that certain combinations of high alkali cements and siliceous aggregate produced expansion and cracking in field structures and laboratory specimens exposed to moisture. The reaction involves the formation of expansive alkali-silica gels, which then imbibe water, swell, and crack the concrete. Interim solutions used to solve the problem were the restriction of cement alkali levels to a certain maximum, and addition of suitable pozzolanic material to help control the reaction.

What is often called ACR seems to really involve two types of reaction. In the first, included silica in the carbonate is really participating in an ASR reaction, with the resulting expansion and cracking of the concrete, as with ASR. In the second type, there is a reaction of the carbonate aggregate, which usually contains some proportion of the mineral dolomite. The reaction involves the dissolution of dolomite and the formation of brucite, with apparent attendant volume change.

There are a number of recommendations to prevent AAR, including low alkali cements and the addition of pozzolans, silica fume, ground granulated blast furnace slag, and lithium chemicals to the concrete.

A range of tests are available to evaluate materials and concrete for susceptibility to these reactions, however all have certain drawbacks so work continues to develop more definitive tests. ASTM C295, Petrographic Examination of Aggregates for Concrete (14), provides guidance on the identification of potentially reactive aggregates. ASTM C227, Potential Alkali Reactivity of Cement-Aggregate Combinations (15), provides a means to evaluate cement and aggregate in combination prior to use. It has several drawbacks, including the length of time needed to reach definitive results, which may be over a year. In response to this shortcoming ASTM C1260, Potential Reactivity of Aggregates (16), was developed, and this method provides results in 16 days. However, it is an aggregate only test, not a test of cement-aggregate combinations, and some people consider it too severe. The concrete prism test, ASTM 1293 (17), was developed for the evaluation of aggregate, however this is a long term test. ASTM C1105, Length Change of Concrete Due to Alkali-Carbonate Rock Reaction (18), and ASTM C586, Potential Alkali Reactivity of Carbonate Rocks for Concrete Aggregates (Rock Cylinder Method) (19) were developed to evaluate aggregates and concretes for ACR. Test(s) are still needed to provide rapid, definitive evaluation of job mix concretes for their AAR potential.

Much work continues to be done in the subject area of AAR. ACI Committee 221, Aggregates, recently completed a state-of-the-art report on AAR (20), which is available now as a separate, and will be included in the next edition of the ACI Manual of Concrete Practice.

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Factors Interaction

As can be seen by the above discussion of durability factors, each of these factors, even when taken separately, creates a complex set of characteristics and conditions to try and evaluate and interpret in order to judge or predict the durability performance of concrete. However, in actual practice, it is very seldom that only one of these distresses occurs in deteriorating in-service concrete (21). The rate of progress of most of these distress mechanisms is dependant on the permeability of the concrete. As one distress cracks or otherwise “opens up” the concrete, it becomes more susceptible to the other distress mechanisms. By the time concern is raised about the performance of a structure, there are usually several distresses at work, making forensic analysis for determination of the original major cause difficult at best. In order to assist in the conduct of systematic concrete analysis for pavements, the Federal Highway Administration is sponsoring a study being conducted by Michigan Technological University entitled “Detection Analysis and Treatment of Materials-Related Distress in Concrete Pavements”. The objectives of the study are to provide guidelines for: field evaluation and representative sampling of the pavements; laboratory evaluation of the samples in order to determine the cause(s) of the distress; and appropriate treatments of the distress in existing pavement and prevention of the distresses in new pavement. The impetus of the study was several recent examples of distressed concrete examined by different groups, who then reached different conclusions as to the cause of deterioration. With a systematic set of guidelines in place for the forensic process, hopefully more consistent analyses can be reached, even by independent investigations.

SUMMARY

This paper provides an overview of the factors that influence the overall durability of concrete in service. Those of major importance are freeze-thaw cycling; aggressive chemical attack; surface abrasion; corrosion of included steel; and alkali-aggregate reactivity. Although there are a number of tests unique to each aspect of durability, there are some tests and characteristics which usually bear a direct relationship to the overall durability of the concrete, no matter what the factor under consideration. Review of the above sections reveals these characteristics to be low water-cementitious materials ratio; dense concrete with low permeability; and often high (or higher) strength. This is because most of the mechanisms responsible for the various types of deterioration depend on the movement of chemicals in solution or water into or through the concrete in order to produce their detrimental effect. Selecting materials and mix designs and carefully controlling construction in order to get the concrete in place with these characteristics, will go a long way toward insuring the long-term durability of the concrete.

It is seldom that a concrete undergoing deterioration is subject to only one type of distress. This is because many of the mechanisms depend on migration into or through the concrete. As the concrete’s permeability is increased by one type of deterioration, it becomes more susceptible to attack by other mechanisms.

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There are a number of tests specific to each aspect of durability; in many cases new or more refined tests are needed to obtain a definitive evaluation of the potential for the distress with the concrete being used in the exposure in which it will be placed. Systematic guidelines are needed for the forensic examination of in-service concrete; especially since once deterioration starts, there are usually multiple mechanisms at work.

In closing, it should be noted that ACI Committee 201 (Durability of Concrete) is currently developing a revision to 201.2R, Guide to Durable Concrete (3), in order to include the latest information on occurrence, testing, and prevention of distress in concrete structures.

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Comparison of Known and Determined Water-Cement Ratios Using Petrography

by J. J. Liu and M. S. Khan

SYNOPSIS

At present, there are no standardized test methods for the determination of water-cement ratio (w/c) of a specimen of hardened concrete. Various methods that have been used include absorption of a water drop on a concrete surface, resistance of cement paste to scratching, polarized-light microscopy, and optical fluorescence microscopy. In these determinations, usually experience and judgment are heavily relied upon. In the absence of a reliable standardized technique, it is prudent to estimate the w/c by using specimens with known w/c and a combination of several techniques, rather than relying on a single technique. The objective of this paper is to demonstrate the successes achieved by following this approach. Concrete specimens with w/c of 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70 were prepared and analyzed by visual examination, resistance to needle scratching, water absorption, and blue-dyed thin section techniques. This combined technique approach was then used to analyze concrete specimens from the field.

Keywords: alkali-silica reaction; concretes; deterioration; freezing and thawing; microscopy; petrography; porosity; water-cement ratio

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INTRODUCTION

Water-cement ratio (w/c) is often regarded as the most important parameter that affects both the strength and durability of concrete, and is usually considered as the strongest indicator of concrete quality. For different types of structures and exposures, maximum w/c are specified and mixture proportioning mostly starts with the w/c . As cement and aggregates are mixed with water, the hydration products start filling the originally mixing-water filled space. The amount of space originally occupied by mixing water is related to the w/c of the concrete. The smaller the fractional volume of the mixing water, the lesser the amount of hydration product required to fill it and stronger the concrete (1). As the w/c is decreased, the porosity of the paste is decreased and the concrete becomes more impermeable to moisture entering the concrete. The ingress of moisture in the concrete is the cause of many concrete deterioration problems, including damage caused by freezing and thawing and alkali-silica reaction. The situation becomes even worse when the moisture entering the concrete is contaminated with aggressive chemicals such as chlorides and sulfates, which promote corrosion of reinforcing steel and sulfate attack (2). The importance of proportioning mixtures with a low w/c and maintaining a low w/c has been highlighted in numerous publications, including the ACI 201 "Guide to Durable Concrete" (3).

However, in practice, the specified w/c is often exceeded due to various reasons. Sometimes water is added at the job site to increase workability which results in a more porous cement paste and consequently to a lower concrete strength and durability. An unexpected rainstorm during or immediately after the placement of the concrete may increase the w/c in the surface layer of concrete. A concrete mixture with non-uniform mixing and compaction results in a non-uniform w/c , introducing weak zones in the concrete. The surface layer of the concrete is generally more vulnerable to an increase in w/c . A weak surface layer containing inadequate entrained air and subjected to deicing salts may experience scaling if subjected to freezing and thawing.

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Thus while troubleshooting concrete problems, the w/c is a parameter that many times provides useful leads to the investigator. This is true for both newly placed concrete and concrete that has been in service for many years. In new construction, a concrete placed with a higher than specified w/c typically starts showing problems after its first winter of exposure and the concerned parties need answers to the problem. In existing structures, an assessment of the overall quality of concrete is required for selecting an appropriate repair or rehabilitation system.

Petrography is the most commonly used technique for determining the overall quality of the concrete and also its individual constituents (4). It is also used to identify the characteristic consequences of the various deterioration mechanisms in the concrete. Typically a petrographic analysis involves the study of a lapped section under a stereoscopic microscope with reflected light and a thin section, of the order of 30 μ m thick, under polarized-light microscope with transmitted light. Although petrography has been applied to concrete since 1910s (5), there are no standardized test methods for the determination of w/c in hardened concrete. Various methods have been proposed and applied for the determination of w/c. Using a lapped section and a stereoscopic microscope, the w/c is commonly estimated by studying the paste appearance, paste quality, resistance of cement paste to scratching, and the absorption of a water drop on a concrete surface (6). Another common method is to compare the brightness of the cement paste in a thin section under a polarized-light microscope (7). These techniques are economical and easy to use; however, they rely heavily on experience and subjective judgment.

The most quantitative method to estimate w/c in a hardened concrete specimen is examination of a fluorescent dye impregnated thin section under a microscope with an ultraviolet light illuminator. It has been found that the gray level of the illumination is proportional to the w/c in the concrete (8,9). The method is not popular, primarily due to expensive equipment involved. Also, it reflects the w/c at discrete points of the thin section.

In the absence of a reliable standardized technique for the determination of w/c, it is prudent to estimate the w/c by using specimens with known w/c and a combination of techniques, rather than relying on a single technique. The objective of this study is to demonstrate the successes achieved by following this approach. Concrete specimens with varying w/c ratios of 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.6, 0.65 and 0.70 were studied using stereoscopic microscope techniques, including visual examination, resistance to needle scratching, and absorption of a water drop. These investigations were followed by the study of concrete thin sections, impregnated with blue epoxy.

Although it is not common to estimate paste porosity in a concrete specimen using blue-dyed epoxy, this technique has been widely used to estimate the porosity and permeability of sandstone and other media in the petroleum

Industry (10). This combined technique approach was then applied to estimate w/c of concrete samples from structures in the field, without knowing the intended value. The estimated w/c were generally consistent with the intended values.

THEORETICAL CONSIDERATIONS

The techniques used in this study for the estimation of w/c are based on the cement paste porosity characteristics. The pore space of the concrete paste directly controls the paste water absorption, paste hardness, paste appearance, and the amount of epoxy resin that can be impregnated into the paste.

There are two different types of pore spaces in the cement paste. The first type is the capillary pore, which is the remnant of the originally mixing-water filled space between the cement particles. The second type of pore space is the gel pore, which is trapped in the hydrated cement paste structure, C-S-H (11). The capillary pores are generally 3.2 to 3,000 nanometers in diameter, whereas the gel pores are less than 3.2 nanometers in diameter (12). Water trapped in the capillary pores is freezable and evaporable, whereas only part of the water trapped in gel pores is evaporable and none is freezable under normal conditions. The capillary porosity is determined by w/c and the degree of hydration, as given by the equation of T.C. Powers (11):

$$P_c = w/c - 0.36\alpha \quad (1)$$

Where P_c is the capillary porosity, and α is the degree of hydration. Therefore, with known degree of hydration, w/c will be determined if the capillary porosity is measured.

The capillary porosity can be measured empirically by comparing the paste properties with concrete samples of known w/c. The water absorption rate, the softness of the paste, and the amount of epoxy impregnated, are proportional to the porosity of the cement paste.

EXPERIMENTAL PROGRAM

Concrete mixtures with w/c of 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.6, 0.65 and 0.70 were prepared and specimens were cast. ASTM Type I portland cement, ASTM No. 57 coarse aggregates, and Virginia "A" sand were used in these concrete mixtures. The proportions of different constituents are shown in Table 1. The gradings of the coarse and fine aggregate are shown in Table 2 and Table 3, respectively. The coarse aggregate had a specific gravity of 2.6 and a water absorption of 0.6%, and was composed of granite and granite gneiss. The fine aggregate had a specific gravity of 2.62 and water absorption of 0.6%, and was composed of mostly quartz and small amounts of chert and feldspar.

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An air-entraining admixture was used in all concrete mixtures. A high-range water-reducing admixture (HRWRA) was used in the mixtures with w/c of 0.30 and 0.35 in order to obtain a reasonable workability. After 24 hours of air curing, the specimens were demolded and immersed in lime saturated water at 35°C for a period of 7 days.

After 7 days of curing, lapped and thin sections of the concrete samples were prepared for petrographic examination. The lapped section was prepared by placing the specimen with water and silicon carbide abrasive powder, on vibrating lap equipment, for 8 hours. The lapped section was then cleaned and air-dried overnight before analyzing. The paste appearance, hardness, and paste reaction to needle scratching were studied by examining lapped sections under a stereoscopic microscope. A surgical needle was used to scratch the paste and to apply water drops onto the lapped surface. Also, for each specimen, a thin section impregnated with blue epoxy was made. The porosity and amount of blue dye impregnated into the paste were examined under a polarized-light microscope.

TEST RESULTS AND DISCUSSION

The characteristics of the concrete specimens of different w/c investigated by different techniques are discussed below. These specimens were moist cured for a period of 7 days at a temperature of 35°C. The cement hydration at the time of analysis was estimated to be approximately 80%, by studying the unhydrated cement particles in the paste under the polarized-light microscope.

Paste Appearance and Resistance to Needle Scratching

The lapped concrete samples (see Figure 1) examined under stereoscopic microscope had three distinct ranges of paste characteristics. In the specimens with w/c of 0.30, 0.35 and 0.40, the paste appeared dense and dark. It was hard and had a smooth ceramic-like appearance. The cement paste was difficult to break using a needle. In the specimens with w/c of 0.45, 0.50 and 0.55, the paste was still hard but was relatively easy to break with a needle. The specimens with w/c of 0.60, 0.65 and 0.70 had a loose cement paste with a sugary texture. The paste was soft and easy to break using a needle. When broken, the paste tended to curl and had feathery edges. As the w/c of the specimens increased, the paste color became lighter and lighter.

Absorption of a Water Drop

A surgical needle was used to drop water on the lapped surface of the concrete section. The reaction of the water drop was observed and the stand-up time was measured. On the concrete surfaces with w/c of 0.30 and 0.35, the water drop stood up like that on a ceramic surface and appeared rolling. It did not begin to spread out until after 7 to 8 minutes. The water drop disappeared after an average of 11 to 14 minutes, on 10 measurements. For concretes with w/c of 0.40 and 0.45, the water drop stood up but started to spread out after 3 to 4

minutes and disappeared after an average of 5 to 6 minutes. On concrete surfaces with w/c of 0.50 and 0.55, water drops quickly spread out and were completely absorbed after 4 to 5 minutes. Concrete surfaces with w/c ratios of 0.60, 0.65 and 0.70 could barely hold the water drop and it disappeared quickly at an average of 2 to 3 minutes.

Figure 2 shows a plot of the stand-up time of the water drop with w/c; each data point represents an average of 10 measurements. The following relationship with a correlation coefficient (R^2) of 0.96 was obtained:

$$y = 0.9961 x^{-0.4485} \quad (2)$$

Where, y is the w/c and x is the stand-up time, in minutes, of the water drop on the concrete surface.

Examination of Thin Section under Polarized-Light Microscopy

When examined under polarized-light microscope, concrete pastes with different w/c showed distinctive characteristics. The amount of blue epoxy impregnation appeared to be directly related to the w/c of the concrete. The paste in the specimens with w/c of 0.30 and 0.35 appeared dark, dense, and almost non-transparent. Very little blue epoxy was impregnated into the paste (see Figure 3). In the specimens with w/c of 0.40 and 0.45, the paste still looked dark and dense, but impregnated blue epoxy began to show in the paste (see Figure 4). In the specimens with w/c of 0.50 and 0.55, there was substantial increase in the amount of blue-dye impregnation, although the paste still looked dense (see Figure 5). Finally, in the specimens with w/c of 0.60, 0.65 and 0.70, significant amount of pore spaces were visible in the microscope, and the paste was largely impregnated with blue dye (see Figure 6).

Examination of Field Samples

The analysis of the samples with known w/c indicates that the w/c of hardened concrete can be estimated by studying paste properties with stereoscopic and polarized-light microscopes. By combining the examinations of a lapped section and a blue epoxy-impregnated thin section, an estimated w/c range can be obtained. In order to evaluate the accuracy of w/c estimation using these combined techniques, 13 concrete samples, obtained from different type of structures, were analyzed without knowing their intended w/c value. The estimated w/c range was then compared with the intended w/c ratio. For all but two of the 13 samples, the intended w/c ratio was in the estimated w/c range (see Figure 7). Among these two samples, one of them did not achieve the designed compressive strength value. It was concluded that the low compressive strength was most likely caused by a w/c higher than the intended value.

Water-Cement Ratio and Other Durability Parameters 17**SUMMARY AND CONCLUSIONS**

The capillary porosity of the concrete paste directly relates to the w/c in the concrete. The capillary porosity can be evaluated by studying the paste under a stereoscopic microscope and a polarized-light microscope. As the w/c of the concrete specimens is increased, there is a gradual but distinct change in the appearance of the cement paste, from dark and dense to lighter and more porous. The softness increases as the w/c increase, which is reflected in the paste reaction to needle scratching. Paste in a low w/c concrete is hard to break, but when it breaks the broken piece tends to form flakes. Paste in high w/c concrete, however, is easy to break with a needle. The broken pieces usually curl and have feathery edges.

The stand-up time of a water drop on a lapped concrete section has a good correlation with the w/c ratio of the concrete ($y = 0.9961 x^{-0.4485}$). On the surface of low w/c (0.30, 0.35) concrete, the water drop stands up and appears as rolling, whereas concrete of high w/c (0.60, 0.65, 0.70) can barely hold the water drop, which disappears quickly into the paste. On lapped surfaces of concrete with intermediate w/c (0.40, 0.45, 0.50, 0.55), water drop stands up but spreads faster than that on the concrete surface of low w/c.

The data presented here on water drop technique have been obtained on concrete specimens with approximately 80% of hydration, by using water as a lapping medium. In concrete specimens with lesser degree of hydration, the lapping process may contribute to further hydration of the specimens and w/c estimates could be less conservative. Also, the type of coarse and fine aggregates would influence the relationship between w/c and stand-up time of water drop, to some extent. Considering these sensitivities of the water drop technique, the technique should be used in conjunction with other techniques, as described in this paper, not as a stand-alone technique.

The amount of blue epoxy impregnated in the thin section is an effective indicator of porosity and thus the w/c. For low w/c concrete, paste is dense and dark with very little blue epoxy impregnated. With the increase of w/c ratio, more and more blue epoxy is impregnated into the paste, which can be clearly seen under a polarized-light microscope.

Results of 13 field samples analyzed suggest that by using samples of known w/c and these techniques together, a reliable estimate of the w/c of the concrete can be made. The reliability of w/c estimates using this combined technique approach would be improved by studying specimens of known w/c, with as many combinations of mixture proportions as possible. This would include concrete mixtures prepared with different cements, aggregates, and mineral admixtures such as fly ash, silica fume and slag.

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TABLE 1—CONCRETE MIXTURE PROPORTIONS.

Mixture	Coarse Aggregate kg/m ³	Fine Aggregate kg/m ³	Cement kg/m ³	Water kg/m ³	w/c
#1	980.4	653.6	570.0	171	0.30
#2	1029.2	686.2	488.6	171	0.35
#3	1065.9	710.6	427.5	171	0.40
#4	1094.4	729.6	380.0	171	0.45
#5	1117.2	744.8	342.0	171	0.50
#6	1135.9	757.2	310.9	171	0.55
#7	1151.4	767.6	285.0	171	0.60
#8	1164.5	776.4	263.1	171	0.65
#9	1175.8	783.9	244.3	171	0.70

TABLE 2—COARSE AGGREGATE GRADING.

Sieve Size	Percentage Retained
37.5 mm	0.0
25.0 mm	5.0
19.0 mm	18.2
12.5 mm	75.3
9.5 mm	95.5
4.75 mm	98.9
2.36 mm	99.0

TABLE 3—FINE AGGREGATE GRADING.

Sieve Size	Percentage Retained
9.5 mm	0.0
4.75 mm	1.3
2.36 mm	15.6
1.18 mm	34.7
600 mm	56.7
300 mm	76.3
150 mm	97.9

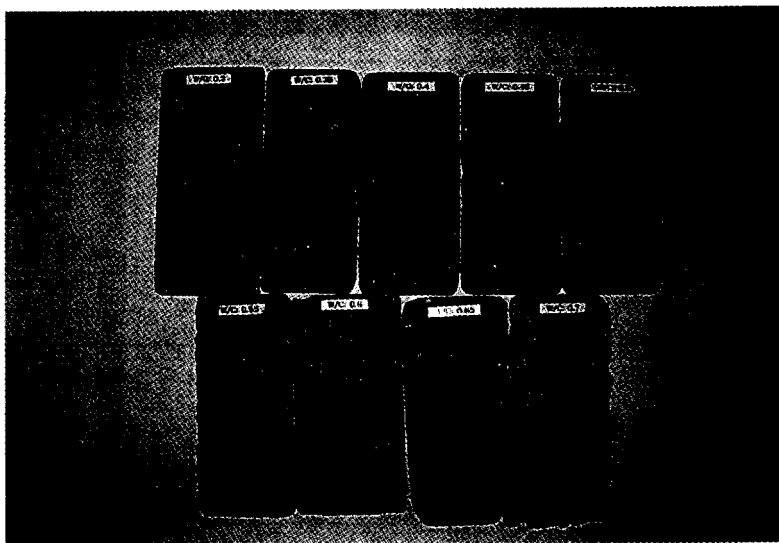


Fig. 1—Lapped sections of concrete specimens with different w/c .

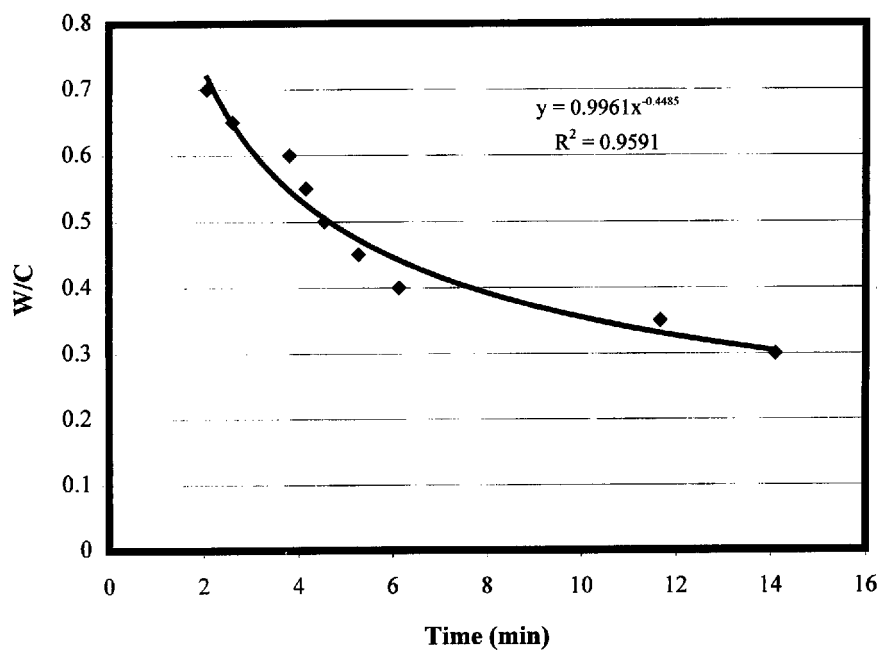
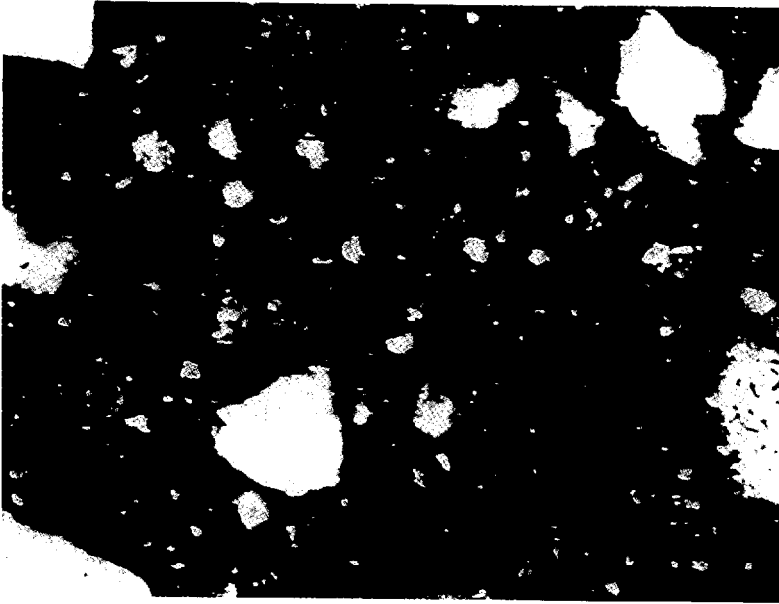
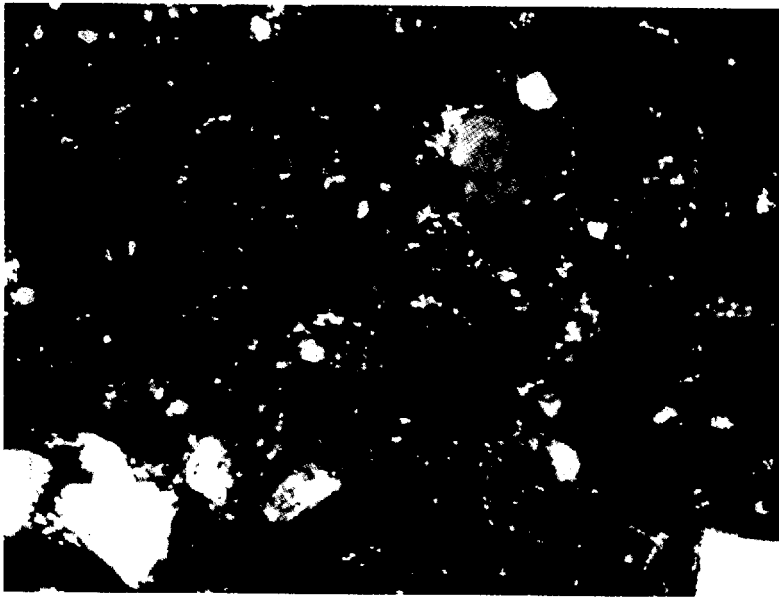


Fig. 2—Relationship between w/c and standup time of water drop.

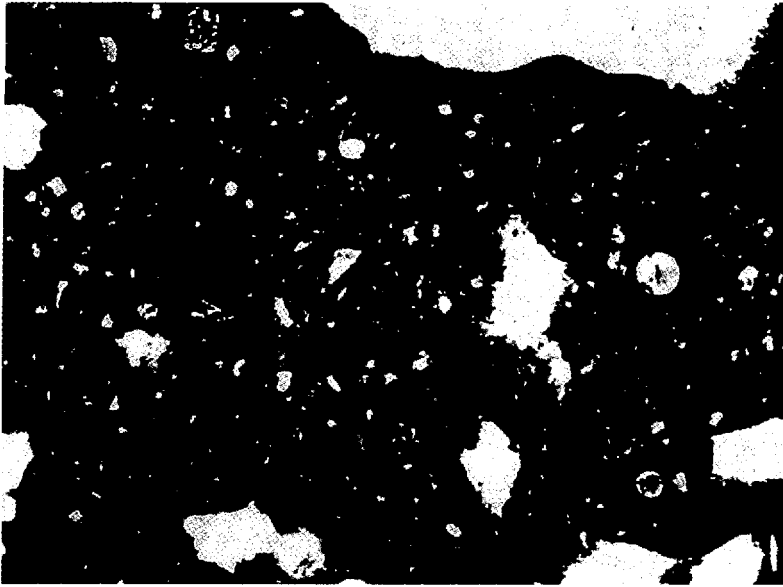


(a)



(b)

Fig. 3—Photomicrographs of low w/c (0.35) concrete specimen with photographed length of 1.4 mm: (a) plane light; and (b) cross-polarized light.

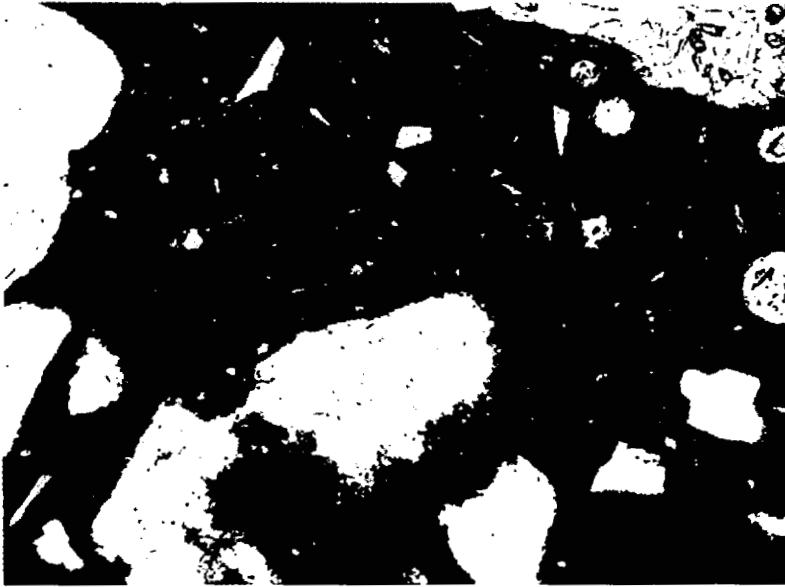


(a)



(b)

Fig. 4—Photomicrographs of low-to-intermediate w/c (0.40) concrete specimen, under plain light: (a) photographed length = 1.4 mm; and (b) photographed length = 0.175 mm.



(a)

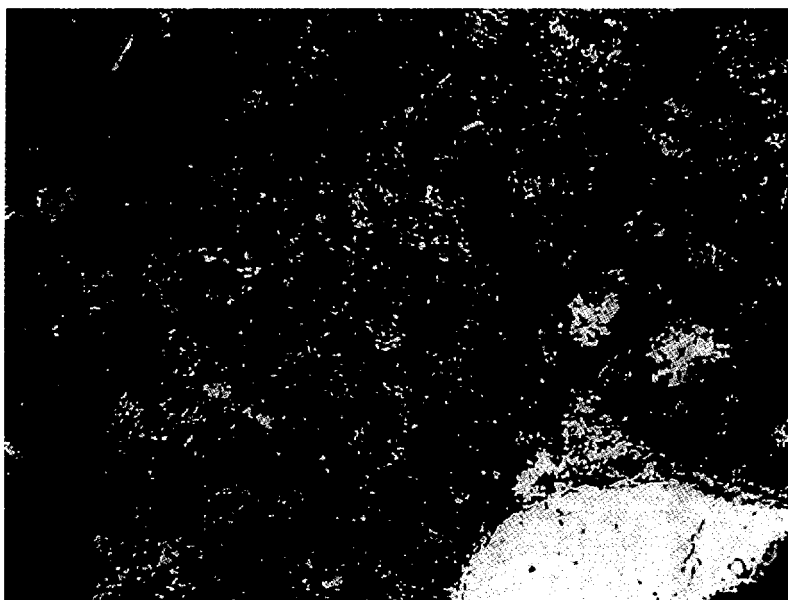


(b)

Fig. 5—Photomicrographs of intermediate w/c (0.50) concrete specimen under plane light: (a) photographed length = 1.4 mm; and (b) photographed length = 0.35 mm.



(a)



(b)

Fig. 6—Photomicrographs of high w/c concrete specimens under plane light, and with photographed length of 0.35 mm: (a) $w/c = 0.6$; and (b) $w/c = 0.70$.

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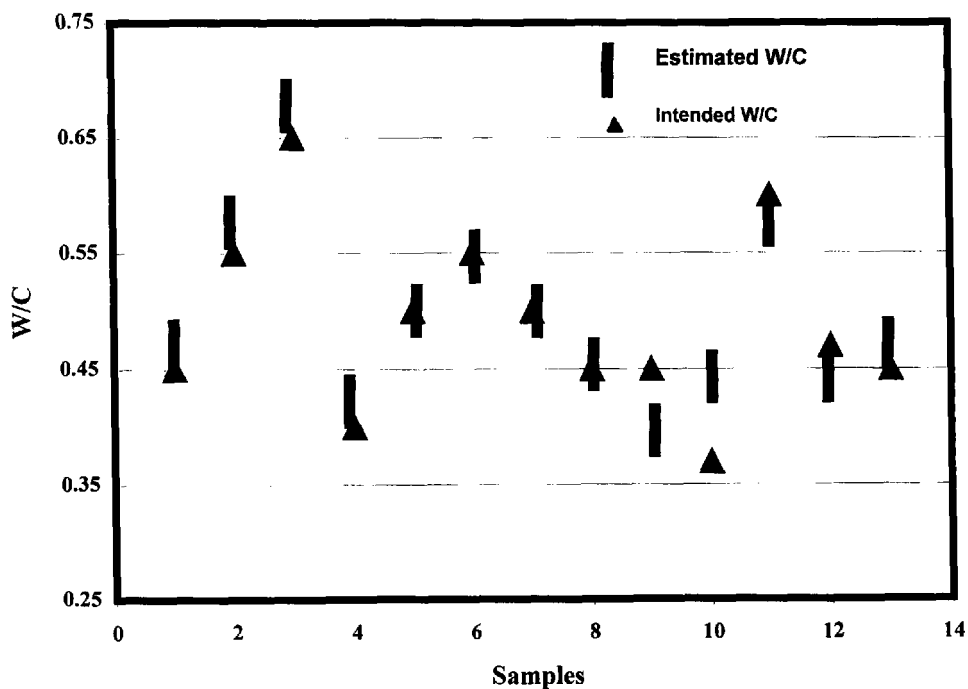


Fig. 7—Comparison between estimated and intended w/c of field samples.

SP 191-3

Determination of Water-Cement Ratio in Hardened Concrete by Optical Fluorescence Microscopy

by U. H. Jakobsen, P. Laugesen, and N. Thaulow

SYNOPSIS

At present, there are no standardized test methods for the determination of water-cement ratio (w/c) of a specimen of hardened concrete. Various methods that have been used include absorption of a water drop on a concrete surface, resistance of cement paste to scratching, polarized-light microscopy, and optical fluorescence microscopy. In these determinations, usually experience and judgment are heavily relied upon. In the absence of a reliable standardized technique, it is prudent to estimate the w/c by using specimens with known w/c and a combination of several techniques, rather than relying on a single technique. The objective of this paper is to demonstrate the successes achieved by following this approach. Concrete specimens with w/c of 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70 were prepared and analyzed by visual examination, resistance to needle scratching, water absorption, and blue-dyed thin section techniques. This combined technique approach was then used to analyze concrete specimens from the field.

Keywords: field tests; fluorescence; microscopy; vacuum forming; water-cement ratio

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INTRODUCTION

The w/c is generally recognized as one of the most important mixture design parameters for concrete, because the w/c effects the strength and durability of concrete. It is easy to control the w/c in the concrete-making process. In hardened concrete it is not possible to determine the w/c directly. There is a need for an indirect, precise method for determination of the w/c in hardened concrete both for quality control in young concrete and in the forensic determination of deteriorated concrete. Optical microscopy of fluorescent impregnated thin sections has proven to be such a method.

Optical microscopy offers a large field of view that facilitates an excellent understanding of the microstructure and the possible causes of production faults or deterioration mechanisms. Furthermore, it is fast and a fairly low cost type of examination. The information obtained from the optical microscope is markedly enhanced by adding a fluorescent facility to the two ordinary light modes namely plane polarized and crossed polarized light. The fluorescent illumination gives information about the capillary porosity (w/c), transition zone porosity, cracks, crack pattern, air void system, porosity of aggregates and paste defects, information necessary for evaluating the quality and the condition of the concrete.

Optical fluorescence microscopy of concrete has been a principal tool in examination of hardened concrete in Denmark for many years. The method is accepted as a NORDTEST Standard since 1991 (1) and used for quality control as well as for evaluating deteriorated concrete. The microscopic examination for quality control follows a Danish Standard procedure, the TI-B 5 (2), and the accredited laboratories performing analyses participate in an annual Round Robin test. The microstructure of the concrete is examined according to the standard procedures.

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For mature concrete the degree of hydration is high and there is a good correlation between the capillary porosity of the cement paste and the w/c as shown in Table 1. The yellow light generated in the fluorescent epoxy has a visual intensity, which is proportional to the amount of fluorescent epoxy present in the cement paste. The intensity can therefore be used as a direct measurement for the capillary porosity and hence the w/c. The change in capillary porosity with w/c is more pronounced at lower w/c than at higher w/c (Table 1). It is therefore much easier to see the change in fluorescence intensity in going from w/c 0.40 to 0.45 than from 0.60 to 0.65. By comparing with standards of known w/c the w/c of an unknown concrete sample can be determined visually quite accurately or measured by automatic image analysis.

Visually determination of the w/c by optical fluorescent microscopy has been described in several papers since 1980 (5, 6, 7, 8, 9, 10, 11, 12, 13). The method has also been used in conjunction with image analyses in the IA-Project (14) and by Elsen et al. (15), Gran (16) and Jakobsen et al (17).

METHODS

Fluorescence Impregnation Technique

The w/c determination of hardened concrete is performed microscopically using fluorescent thin sections. To prepare fluorescent thin sections the concrete has to be vacuum impregnated with a fluorescent epoxy (18, 19, 20, 21). An example of components shown useful for fluorescent epoxy is given below:

Epoxy Resin:	BY 158 from Ciba Geigy
Hardener:	HY 2996 from Ciba Geigy
Fluorescent dye:	Epo-dye from Struers

The procedure for mixing the fluorescent epoxy follows two steps:

- 1.28 grams of powdered fluorescent dye are mixed with 98.72 grams epoxy resin to give a total of 100 gram. The solution is stirred for 24 hours. The fluorescent resin shall be kept in a dark place until use.
- 100 grams of fluorescent resin is mixed with 28 grams of hardener immediately before use. Following this procedure the final mixture contains 1% (w/w) fluorescent dye.

During preparation of the fluorescent epoxy, it is important always to use the same concentration of fluorescent dye in the epoxy. Too much or too little of the fluorescent dye will change the intensity of the green shade of the cement paste when observed in the fluorescent light mode and make comparisons to standards uncertain.

Depending on the condition of the concrete core, the first step of impregnation

may be a full size vacuum impregnation of the core with fluorescent epoxy (Fig. 1). A cracked or weak core should always be full size impregnated before any cutting is performed to avoid further cracking during preparation. The vacuum impregnation procedure is described below:

- The sample must be dry before impregnation but should not be exposed to temperatures above 30-35 °C.
- The sample is wrapped in a strong plastic bag and placed in the desiccator and evacuated for approximately 2 hours. A pump giving a specified maximum vacuum corresponding to a pressure of 3×10^{-2} mbar is suitable for evacuation.
- Fluorescent epoxy is sucked into the evacuated desiccator (through a tube to the bottom of the sample) until the sample is completely covered. The vacuum is released after 3-5 minutes.
- The sample in its plastic bag is removed from the vacuum chamber and placed in a suitable beaker of water to cool, preventing excess heating from the hardening of the epoxy. Care is taken not to mix any water into the epoxy.
- The epoxy is hardened after about 15 hours at 20-25 °C.

After hardening of the epoxy, the sample is cut and features such as cracks can be studied macroscopically using UV-light (Fig. 1). Details of this method have been discussed by Henrichsen & Laugesen (22) and Jakobsen (13). After this step, the sample is ready for further cutting for thin sectioning.

Fluorescent Thin Section Preparation

The thin sectioning can be described in five steps as illustrated in Fig. 2:

Step 1 The concrete sample is cut into small blocks corresponding to the size of the final thin section (e.g. 35 x 45 mm and a thickness of at least 20 mm).

Step 2 One side of the sample piece is glued onto a piece of window glass as reference plane. The opposite side is ground and lapped until final thin section quality before the next impregnation step. The following procedure is used for impregnation:

- ◇ The sample piece is placed in pure alcohol for approximately 15 seconds and dried in an oven for about 1 hour at a maximum temperature of 35 °C.
- ◇ The sample is placed in a suitable beaker, placed in the desiccator and evacuated for approximately 2 hours.
- ◇ Fluorescent epoxy is added without vacuum release and the vacuum is released after 3-5 minutes.
- ◇ The sample is left for another 10 minutes and then removed from the epoxy.
- ◇ The impregnated sample is covered by a piece of strong plastic film in order to minimize the amount of excess epoxy left on the surface.
- ◇ The epoxy is generally hardened after 15 hours at 25 °C.

After hardening, the excess epoxy and further 0.005 mm of the ground concrete surface is ground away. Plane parallel grinding is of utmost importance in this step. The impregnation of the capillary porosity is checked by use of a stereo

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microscope with UV-light mode.

Step 3 The impregnated re-ground surface is glued onto an object glass using UV-hardening glue.

Step 4 The concrete with the window glass attached is cut off leaving approximately 0.5 mm attached to the object glass for thin sectioning. The thin section is then ground and polished to a thickness of 0.020 mm (20 μm).

Step 5 Finally a cover glass is glued onto the ground surface using UV-hardening glue.

Minor refinements to the method described above may be necessary due to the use of different lapping equipment.

The thickness of the finished thin section is checked by the interference colors of quartz in crossed polarized light. With a thickness of 20 μm of the section, the color of quartz is white to gray. A thickness of 25-30 μm can be accepted as long as the reference samples are of the same thickness. However, for other petrographic purposes (such as cement type determination and analysis of powder dispersion) the standard thickness of 20 μm has proved to be superior.

Importance of Proper Impregnation and Regrinding

The general impregnation depth of the fluorescent epoxy into the work piece is dependent on the capillary porosity of the cement paste (23). Fig. 3 shows an example of the impregnation depth of 2 samples, one with a w/c of 0.40 and one with w/c 0.60. As seen from the sketch, the impregnation depth is rather restricted and it is therefore important to place the thin section as close to the impregnated surface as possible to get a fully impregnated thin section. However, it is likewise important to regrind sufficiently to remove excess epoxy on the surface of the sample to prevent 'false' porosity appearance.

The restricted impregnation depth calls for great accuracy in re-grinding the impregnated sample. If the re-grinding depth is too large, the cement paste in the finished thin section is not impregnated through its full thickness. Likewise, if the re-grinding is not parallel to the impregnated surface, the 'deeper lying part' of the thin section may not be well impregnated.

The impregnation depth has been measured in a series of impregnated and cross-sectioned work pieces from concrete, ranging in w/c from 0.35 to 0.70. The impregnation depths are given in Table 2. As seen from the table the process of impregnation and re-grinding is of outmost importance for the quality of the thin section especially when preparing thin sections of concrete with low w/c. The impregnation depth of a sample with a w/c of 0.35 is only 0.020-0.050 mm. It is worth mentioning when impregnating concrete that various highly permeable features such as larger crack systems, aggregate de-bonding, interconnected porosity, and agglomeration of air voids are readily impregnated. Hence, these act as 'impregnation surfaces', causing the cement paste to be impregnated to larger depths through them. Such causes of increased impregnation depth were not present in the samples of Table 2.